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(54) **VALVE-CELL VACUUM DEPOSITION APPARATUS INCLUDING A LEAK DETECTION DEVICE AND METHOD FOR DETECTING A LEAK IN A VACUUM DEPOSITION APPARATUS**

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See application file for complete search history.

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(57) **ABSTRACT**

The leak detection device is adapted to test the tightness of an inner tank (24) of a valve cell (20) of the vacuum deposition apparatus, either at its filling flange (25) or at its inner tank valve (28). A vacuum deposition apparatus equipped with a helium detector (51) mounted as a by-pass of the output of a high-flow-rate turbomolecular pump (42) which is connected to the vacuum deposition chamber (30) of the vacuum deposition apparatus by a slide gate valve (43). A valve-cell vacuum deposition apparatus equipped with a helium-based leak detection device including gas injection elements (52, 53) adapted to inject a gaseous mixture into the outer enclosure, the gaseous mixture being consisted of pure helium and an inert gas, and a method for detecting a leak in a valve-cell vacuum deposition apparatus are also described.

20 Claims, 3 Drawing Sheets

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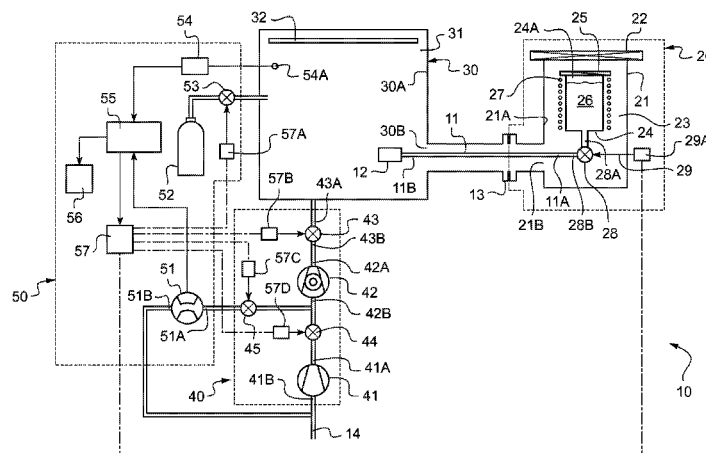
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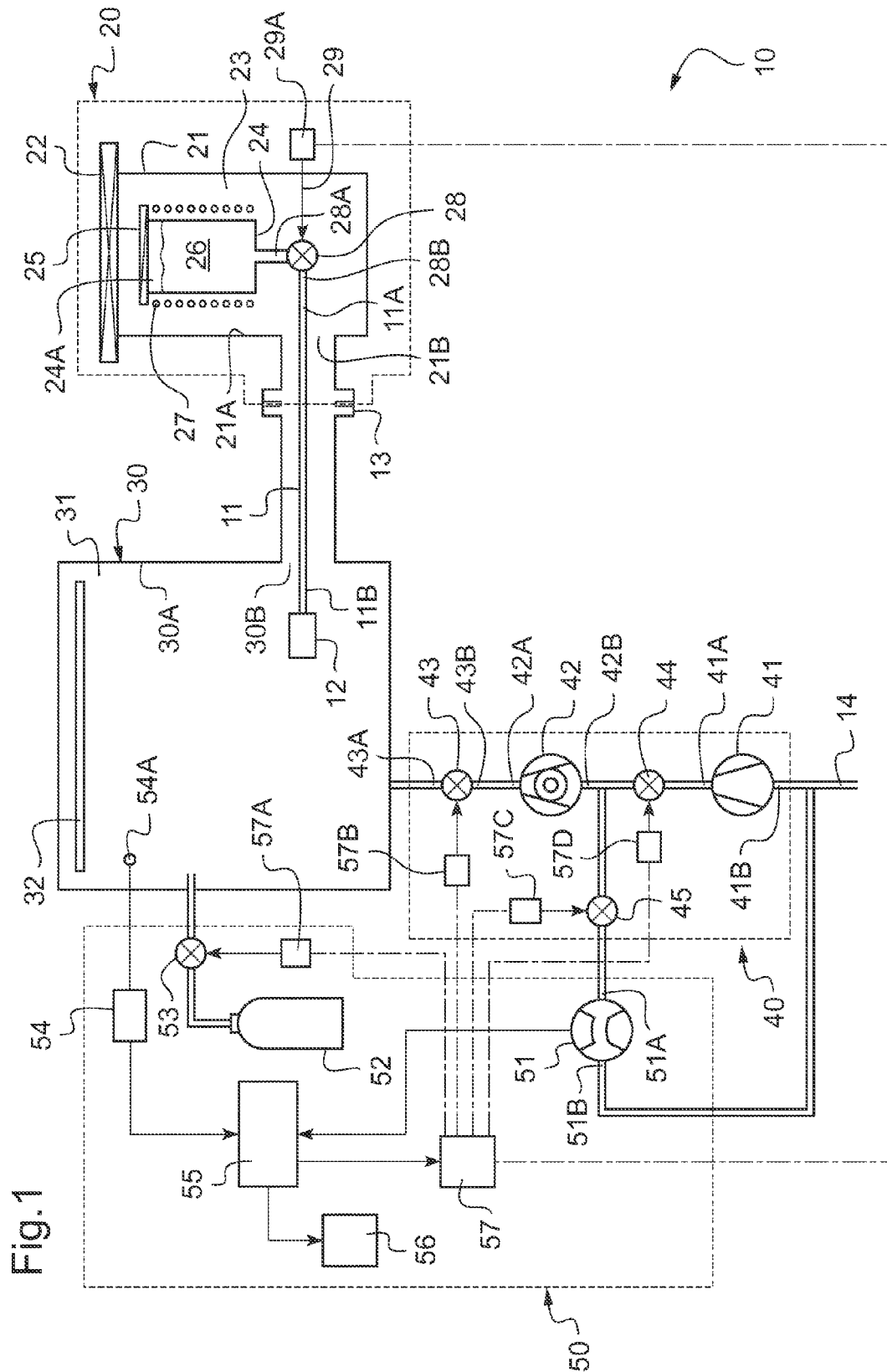
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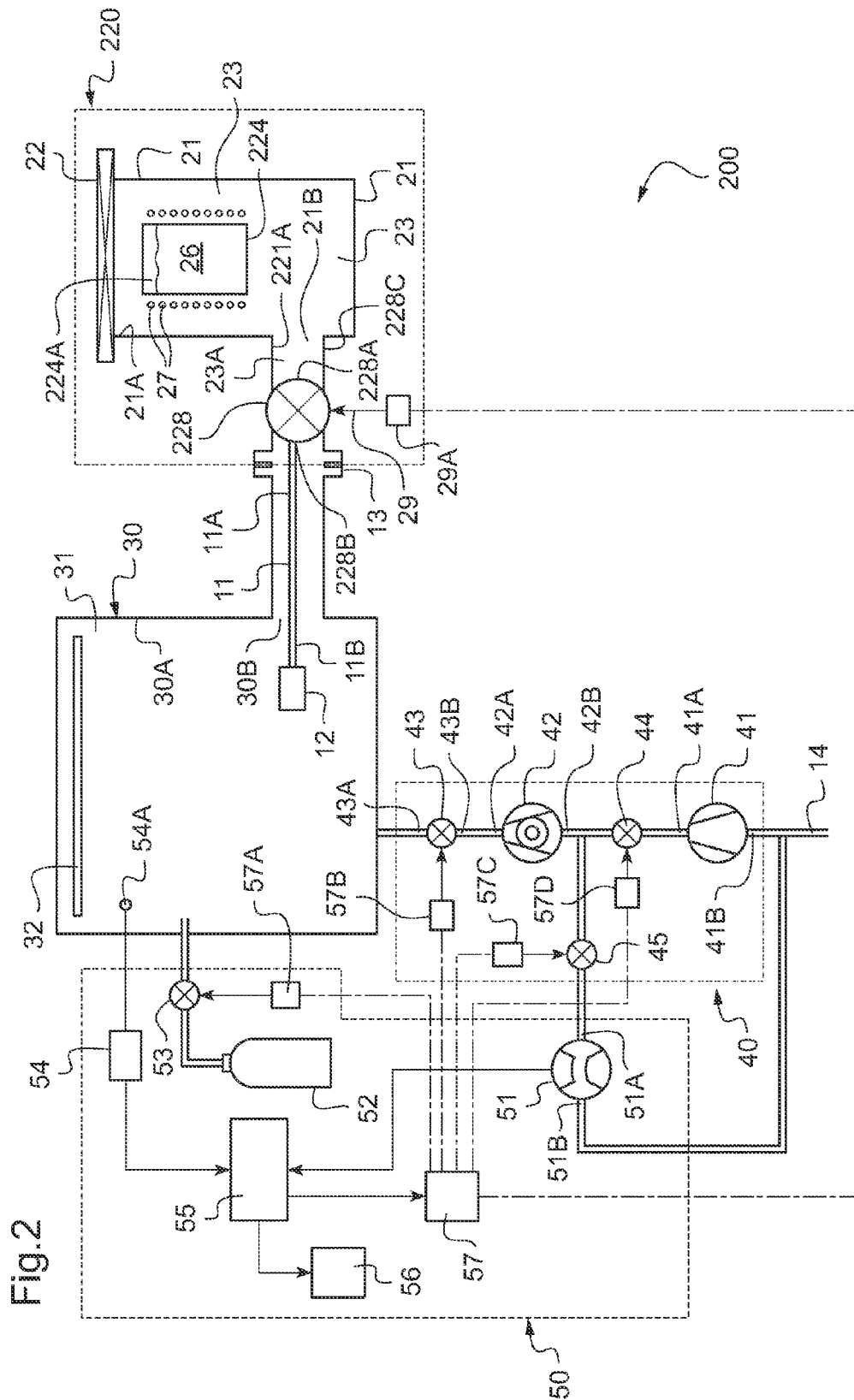
CPC G01M 3/20; G01M 3/226; G01M 3/202; C23C 16/52; C23C 14/564; C23C 14/243; F22B 1/066

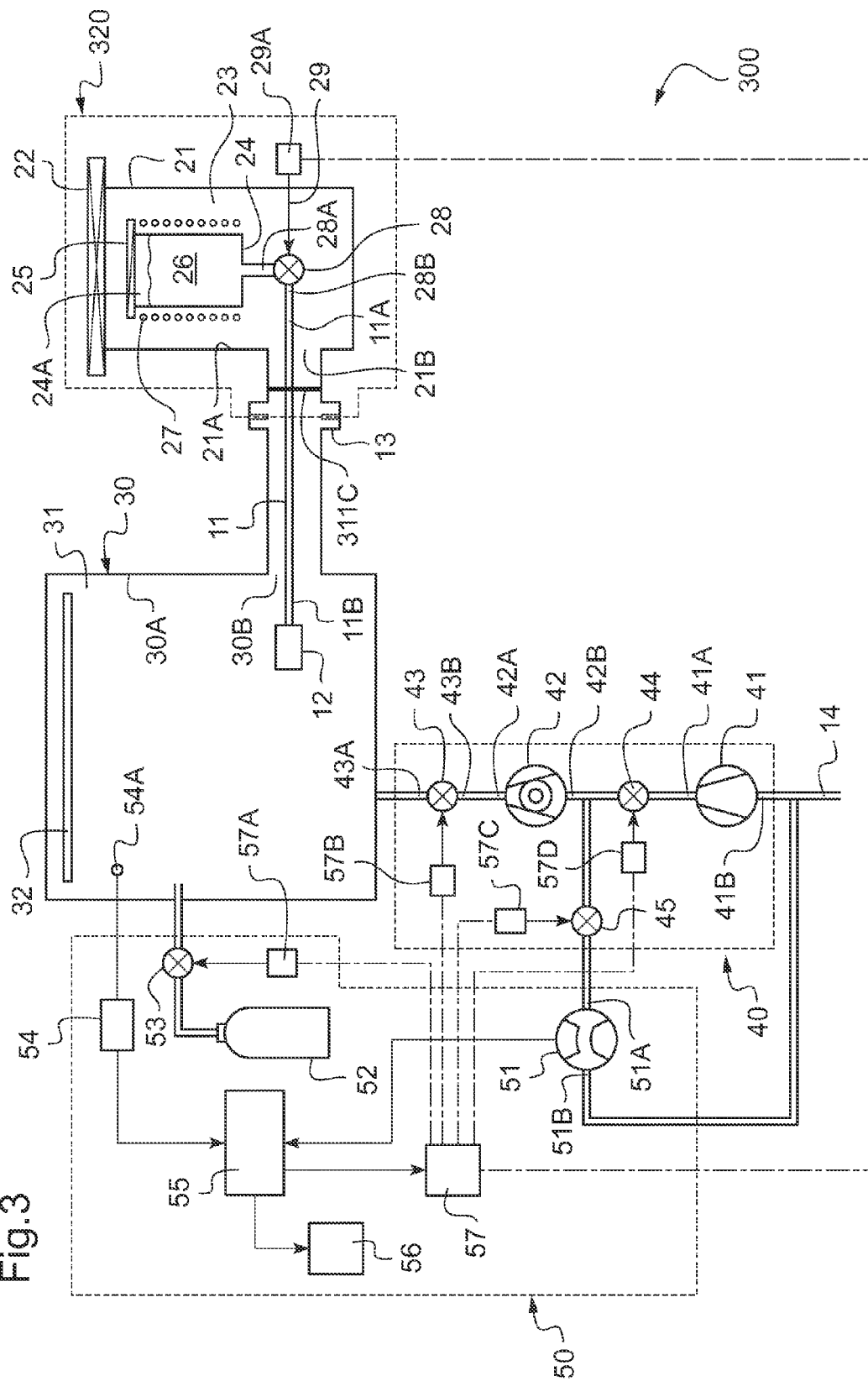


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**VALVE-CELL VACUUM DEPOSITION
APPARATUS INCLUDING A LEAK
DETECTION DEVICE AND METHOD FOR
DETECTING A LEAK IN A VACUUM
DEPOSITION APPARATUS**

FIELD OF THE INVENTION

The invention relates to a vacuum deposition apparatus comprising a valve cell, a transfer tube and a vacuum deposition chamber.

The invention more particularly relates to a vacuum deposition apparatus provided with a helium-based leak detection device.

BACKGROUND OF THE INVENTION

Vacuum deposition apparatuses are widely used in the industry to deposit layers, sometimes very thin, of materials on substrates of various sizes.

It is known in the prior art that a valve-cell vacuum deposition apparatus includes:

at least one valve cell comprising:

an outer enclosure,

an inner tank located inside the outer enclosure and adapted to contain a material to be evaporated,

an inner tank valve comprising a valve input port in fluidic communication with the inner tank and a valve output port,

a vacuum deposition chamber,

a transfer tube comprising a tube input port connected to the valve output port and a tube output port located inside the vacuum deposition chamber, the tube output port including an injector adapted to generate a jet of vapour of the material to be evaporated inside the vacuum deposition chamber, the vacuum deposition chamber being connected to the outer enclosure by means of a connecting flange, and

pumping means adapted to evacuate the inside of the vacuum deposition chamber, the pumping means comprising a primary pump having a primary pump input and a primary pump output, and a secondary pump having a secondary pump input and a secondary pump output.

The industry of microelectronics uses for example such a vacuum deposition apparatus to deposit materials using techniques such as Molecular Beam Epitaxy (MBE) or Molecular Beam Deposition (MBD). Conductive materials (copper, zinc, nickel, chromium, for example), isolative materials (oxides, nitride, for example), or semi-conductor materials (silicon, germanium, arsenic, phosphorus, antimony, gallium, indium, aluminium, for example) are deposited this way on substrate of silicon, germanium or other suitable materials.

The materials, either elemental or compound, and in particular semi-conductor materials, which are used in a very pure form in such vacuum deposition apparatuses, are stored in high-volume closed inner tanks for ensuring a continuous operation of these apparatuses during long periods and optimizing the costs of production of the semi-conductor components manufactured. The price of these materials being high, it is necessary to avoid the unnecessary loss or the irreversible degradation of all or part of the volumes stored in the inner tanks.

Therefore, the inner tanks of materials must be sealingly closed so as, for example, to prevent any leak of vapour of the material to be evaporated toward the outside of the inner tank or to prevent any oxidation or other chemical reaction with

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another compound (water, for example) entering into the inner tank. This makes it possible not to lose the content of the inner tank, which would cause additional costs of exploitation.

Likewise, the inner tank valve must be tight, that is to say that in its "closed" position, it totally blocks the flowing of the material to be evaporated between the inner tank and the vacuum deposition chamber. This makes it possible in particular not to pollute the vacuum deposition chamber with the material to be deposited when it is not desired, which would cause problems, for example when depositing another material on the substrate.

The vacuum deposition apparatuses according to the prior art do not make it possible to verify rapidly and in any circumstances the tightness of the inner tank or the inner tank valve, in particular in operation or during the use of pyrophoric compounds.

Leak detection systems are known from the prior art, which make it possible to test the tightness of the other members of the apparatus, such as the outer enclosure or the vacuum deposition chamber, for example.

SUMMARY OF THE INVENTION

In order to remedy the above-mentioned drawback of the prior art, the present invention proposes a valve-cell vacuum deposition apparatus equipped with a leak detection device for testing the tightness of the inner tank and/or the inner tank valve, with a short response time and a high measurement dynamics.

For that purpose, the invention relates to a valve-cell vacuum deposition apparatus as described in the introduction, the vacuum deposition apparatus being characterized in that it includes a leak detection device comprising:

gas injection means adapted to introduce helium in the vacuum deposition chamber,

gas pumping means adapted to pump the helium present in the vacuum deposition chamber,

a helium detector comprising a detector input, the helium detector delivering a signal representative of the helium flow rate through it,

pressure measuring means adapted to measure the pressure inside the vacuum deposition chamber, and

signal processing means adapted to process the signal relating to the helium flow rate delivered by the helium detector and the signal delivered by the pressure measuring means, to deliver a signal representative of the leak rate of the inner tank and/or the inner tank valve, and in that the pumping means also include:

a secondary pump valve placed between the vacuum deposition chamber and the secondary pump input, for example via a high-conductance path,

a primary pump valve placed between the secondary pump output and the primary pump input,

a by-pass valve placed between the secondary pump output and the input of the helium detector, the by-pass being made upstream the primary pump valve.

The vacuum deposition apparatus according to the invention is so arranged that it is possible:

to close the primary pump valve when the by-pass valve is open, the compressed gases coming out of the secondary pump output being then conducted toward the detector input, and

to close the by-pass valve when the primary pump valve is open, the compressed gases coming out of the secondary pump output being then conducted toward the primary pump input.

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Therefore, the device according to the invention makes it possible to use the combined pumping capacities of the secondary pump and the primary pump to evacuate very rapidly the vacuum deposition chamber, as soon as the secondary pump valve opens, and to discharge this way the major part of the gaseous helium present in the vacuum deposition chamber. Then, this high quantity of helium does not saturate the helium detector of the leak detection device, which usually includes a mass spectrometer.

Moreover, upon opening of the by-pass valve and closing of the primary pump valve, as the helium detector is not saturated with helium, it can perform very rapidly a leak detection test, using all the measurement dynamics of the detector.

That way, a leak of the inner tank valve is detected with a low response time and a good accuracy.

The use of the secondary pump valve makes it possible, when this valve is closed, to separate the vacuum deposition apparatus from the pumping means of the vacuum deposition chamber, these latter being nevertheless capable of operating in continuous so that their pumping capacities are immediately available when the secondary pump valve opens.

Moreover, other advantageous and non-limitative characteristics of the vacuum deposition apparatus according to the invention are the following:

- the signal processing means deliver a signal representative of the leak rate of the inner tank valve and the inner tank; the inner tank valve is located inside the outer enclosure, with its valve input port connected to the inner tank; the tube input port of the transfer tube is located inside the outer enclosure, with the transfer tube going through a first wall of the outer enclosure and a second wall of the vacuum deposition chamber;
- the pressure measuring means deliver a signal representative of the pressure inside the inner tank;
- the outer enclosure is in fluidic communication with the vacuum deposition chamber;
- there are provided isolation means adapted to isolate the outer enclosure from the vacuum deposition chamber; the isolation means comprise the inner tank valve;
- the isolation means comprise a weld between the outer enclosure and the transfer tube;
- the secondary pump is a turbomolecular pump;
- the secondary pump valve is a slide gate valve;
- the secondary pump valve is a butterfly valve or a pendulous valve;
- the gas injection means of the leak detection device are also adapted to introduce an inert gas in the vacuum deposition chamber, the inert gas forming a gaseous mixture with helium;
- the leak detection device also comprises piloting means adapted to control the gas injection means and the opening and closing of the inner tank valve, the secondary pump valve, the primary pump valve and the by-pass valve.

The present invention has several advantages. It allows a very rapid stabilization of the signal delivered by the helium detector, allowing the measurement to be performed before the pressure in the inner tank decreases significantly. It also pushes back the pressure domain in which the pressure decrease in the inner tank can be pertinently linked to a leak of this inner tank.

The vacuum deposition apparatus according to the invention makes it possible to measure precisely the valve conductances of the inner tank valve and of a filling flange of all the valve cells installed on the vacuum deposition apparatus, and this over several decades and within a few minutes.

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The leak detection device may be used on valve cells without delay, in particular in nominal operating temperature and pressure conditions, and without re-aeration of the vacuum deposition chambers.

Most of the existing leak detectors may be used with the vacuum deposition apparatuses according to the invention.

The secondary pump may be installed anywhere on the high-conductance pumping line and not necessarily with a secondary pump installed on the vacuum deposition chamber.

The additional cost for implementing a vacuum deposition apparatus according to the invention is much reduced when the use of a secondary pump and an existing leak detector is possible.

With such a vacuum deposition apparatus, the response time of the tests of external leak of the high-volume vacuum deposition chambers is significantly reduced and the efficiency of the leak research is widely improved.

The invention also relates to a method for detecting a leak in a valve-cell vacuum deposition apparatus, the vacuum deposition apparatus including:

at least one valve cell comprising:

- an outer enclosure,
- an inner tank, located inside the outer enclosure, wherein the inner tank contains a material to be evaporated,
- an inner tank valve comprising a valve input port in fluidic communication with the inner tank and a valve output port,
- a vacuum deposition chamber, and
- a leak detection device,

the leak detection method being characterized in that it includes:

firstly, an initialization step wherein:

- the inside of the vacuum deposition chamber is evacuated, and
- an initial leak flow rate in the vacuum deposition chamber is measured by means of a leak detection device, and

secondly, a measurement step wherein:

- the inner tank valve is open to place the vacuum deposition chamber in fluidic communication with the inner tank, and
- a gaseous mixture is injected in the vacuum deposition chamber, and
- a pressure P_0 inside the inner tank is measured, and
- the inner tank valve is closed, and
- the inside of the vacuum deposition chamber is evacuated down to a predetermined pressure in the vacuum deposition chamber, and
- the vacuum deposition chamber is placed in communication with the leak detection device, and
- a final leak flow rate in the vacuum deposition chamber is measured by means of the helium detection device, and
- a leak rate of the inner tank and/or inner tank valve is deduced therefrom.

Moreover, other advantageous and non-limitative characteristics of the leak detection method according to the invention are the following:

- at the end of the measurement step, the leak rate of the inner tank valve and the inner tank is deduced;
- the inner tank valve of the vacuum deposition apparatus is located inside the outer enclosure, with its valve input port connected to the inner tank.

BRIEF DESCRIPTION OF THE DRAWINGS

Embodiments of the invention will be described in detail with reference to the drawings, in which:

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FIG. 1 is a schematic overall view of a vacuum deposition apparatus according to a first embodiment of the invention;

FIG. 2 is a schematic overall view of a vacuum deposition apparatus according to a second embodiment;

FIG. 3 is a schematic overall view of a vacuum deposition apparatus according to a third embodiment.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

To begin, it is to be stated that, in the following description, the terms "top" and "bottom" are used in relation to the room in which the vacuum deposition apparatus is installed, the top denoting the side directed toward the ceiling of the room and the bottom denoting the side directed toward the floor. Likewise, the terms "lower" and "upper" will denote the sides directed toward the floor and the ceiling, respectively.

First Embodiment

FIG. 1

FIG. 1 shows a vacuum deposition apparatus 10 of the type of those used to implement MBE layer deposition techniques.

This vacuum deposition apparatus 10 includes first of all a source or valve cell 20. It will be considered herein that the vacuum deposition apparatus 10 includes only one valve cell 20. Nevertheless, the invention applies as well to a vacuum deposition apparatus including several valve cells, and the one skilled in the art will be able to make the required adaptations to the invention so that it can be applied to such a type of apparatus.

The valve cell 20 comprises an outer enclosure 21 generally tightly closed on its upper part by a cell flange 22. This cell flange 22 is a so-called "cold" flange insofar as its temperature is close to the ambient temperature outside the outer enclosure 21 and the vacuum deposition apparatus 10. In the standard operating conditions, the cell flange 22 has a temperature comprised between 20° C. and 250° C.

The outer enclosure 21 includes outer enclosure walls 21A that define with the cell flange 22 an outer enclosure volume 23. When the cell flange 22 tightly closes the outer enclosure 21, the pressure inside the outer enclosure 21 is comprised between 10^{-2} and 10^{-3} millibar (mbar).

Upon re-aeration, i.e. when the cell flange 22 is open, the pressure inside the outer enclosure 21 is equal to the pressure inside the room, substantially equal to 1013 mbar (i.e. about 1 atmosphere).

Therefore, as the vacuum deposition apparatus 10 is in ambient conditions where the pressure outside the outer enclosure 21 is substantially equal to 1013 mbar, the cell flange 22 must ensure an extremely high tightness between the outer enclosure volume 23 and the outside of the outer enclosure 21, the pressure difference between this two environments being very high.

The valve cell 20 also includes an inner tank 24 entirely located inside the outer enclosure 21, in the outer enclosure volume 23. The inner tank 24 is generally arranged vertically and tightly closed by a filling flange 25 located on its upper side. The inner tank 24 contains a material to be evaporated 26, partly filling the inner tank 24, leaving a free volume 24A in the top part of the inner tank 24.

In the embodiment shown in FIG. 1, the material to be evaporated 26 is arsenic (chemical symbol As), which is used here in the form of arsenic solids.

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In normal conditions, the pressure in the inner tank 24 is comprised between 0.1 mbar and 10 mbar, the pressure depending on the temperature inside the inner tank 24.

As a variant, the material to be evaporated may be for example in the form of solid ingots.

As another variant, the material to be evaporated may be for example phosphorus, antimony, selenium, sulfur, tellurium, magnesium, or compounds such as cadmium tellurium (CdTe), zinc selenide (ZnSe) or gallium phosphorus (GaP).

According to the materials, these latter may be stored inside the inner tank in solid or liquid form.

The filling flange 25 makes it possible to refill the inner tank 24 when the quantity of material to be evaporated 26 comes to run out in the inner tank 24. To carry out this refilling operation, an operator opens the outer enclosure 21 by removing the cell flange 22 and opens the inner tank 24 by removing the filling flange 25. The operator may then fill the inner tank 24 with a new quantity of material to be evaporated 26. The operator then closes the inner tank 24 by means of the filling flange 25 and closes finally the outer enclosure 21 by means of the cell flange 22.

At the end of a step of filling (or refilling) the inner tank 24, it is necessary to make sure that the filling flange 25 tightly closes the inner tank 24 and that no leak is liable to occur from the inner tank 24 toward the outer enclosure volume 23. It will be seen how the embodiment of the vacuum deposition apparatus 10 according to the invention described hereinafter makes it possible to detect such a leak on the inner tank 24.

The material to be evaporated 26 contained in the inner tank 24 is heated by heating means 27 arranged at the periphery of the inner tank 24. The heating means 27 here comprise heating resistances radiating to the inner tank 24 and therefore increasing the temperature thereof. In operating condition, the inner tank 24 is at a temperature comprised between 50° C. and 600° C.

The pressure inside the outer enclosure 21 being low during the operation of the vacuum deposition apparatus 10, in particular when the heating means 27 radiate heat, the outer enclosure walls 21A remain cold, as the relative vacuum inside the outer enclosure volume 23 limits the heating thereof by gaseous conduction from the heating means 27.

Once the material to be evaporated 26 is in gaseous form thanks to its temperature increase due to heating by the heating resistances 27, the vapours of the material to be evaporated 26 are conducted from the output of the inner tank 24 to an inner tank valve 28.

As shown in FIG. 1, this inner tank valve 28 is located inside the outer enclosure 21 and comprises a valve input port 28A and a valve output port 28B. The valve input port 28A of the inner tank valve 28 is connected to the output of the inner tank 24.

In other words, the valve input port 28A is in fluidic communication with the inner tank 24.

Generally, a flap 29 linked to a piston allows opening or closing the inner tank valve 28, the flap 29 being controlled by means of an actuator 29A located outside the outer enclosure 21. Therefore, the inner tank valve 28 is controllable from the outside.

When the inner tank valve 28 is in open position, it lets the material to be evaporated 26 pass through, the latter being capable of flowing from the valve input port 28A to the valve output port 28B.

When the inner tank valve 28 is in closed position, it normally inhibits any flowing of the material to be evaporated 26 from the valve input port 28A toward the valve output port 28B. In this closed position, the inner tank valve 28 is relatively tight. Nevertheless, it is necessary to verify the tight-

ness of this inner tank valve **28** so as to ensure that no leak is liable to occur, i.e. the leak level is below the tolerable leak threshold.

Such a leak may occur between the valve input port **28A** and the valve output port **28B**. It is then referred to “in line” leak, such leak characterizing the more or less closed position of the inner tank valve **28**.

Another leak may also occur from the inner tank valve **28** toward the outer enclosure volume **23**, and thus between the inner tank **24** and the outer enclosure **21**.

It will be seen how the embodiment of the vacuum deposition apparatus **10** according to the invention described hereinafter makes it possible to detect such leaks on the inner tank valve **28**.

The actuator **29A** allows the precise control of the flap **29** so that the inner tank valve **28** can take several positions, between the two extreme positions, open and closed.

As shown in FIG. 1, the vacuum deposition apparatus **10** also includes a transfer tube **11**, the transfer tube **11** including a tube input port **11A** and a tube output port **11B**.

The tube input port **11A** of the transfer tube **11** is located inside the outer enclosure **21** and is connected to the valve output port **28B** of the inner tank valve **28**.

As shown in FIG. 1, the transfer tube **11** goes through a first wall **21A** of the outer enclosure **21**, at a first opening **21B**, and through a second wall **30A** of a vacuum deposition chamber **30**, at a second opening **30B**, in such a way that the tube output port **11B** of the transfer tube **11** is located inside the vacuum deposition chamber **30**, in a chamber volume **31**.

The vacuum deposition chamber **30** is connected to the outer enclosure **21** by means of a connecting flange **13**. In the particular embodiment of the invention shown in FIG. 1, the connecting flange **13** is such that the outer enclosure **21** communicates with the vacuum deposition chamber **30**, the outer enclosure volume **23** and the chamber volume **31** forming a vacuum deposition apparatus volume.

The outer enclosure **21** is thus here in fluidic communication with the vacuum deposition chamber **30**.

Therefore, in this configuration, at the static equilibrium, the pressure in the outer enclosure volume **23** and the pressure in the chamber volume **31** are identical. Hereinafter, it will thus be referred only to the chamber pressure.

Thus arranged, the transfer tube **11** allows conducting the vapours of the material to be evaporated **26** from the valve cell **20**, and more particularly from the tube input port **11A** of the transfer tube **11** connected to the inner tank valve **28** that is not closed, to the vacuum deposition chamber **30**, thanks to its tube output port **11B**.

The vacuum deposition chamber **30** includes in its upper part a substrate **32** on which layers of different materials are deposited, and in particular layers of the material to be evaporated **26**, which is here arsenic.

For that purpose, the tube output port **11B** of the transfer tube **11** includes an injector **12** whose shape and size are optimized to generate a jet of vapour of the material to be evaporated **26** inside the deposition chamber **30**, the shape, the direction and the intensity of the jet being adjusted so that the desired layer of material to be evaporated **26** is deposited on the substrate **32**.

In a variant of the first embodiment, the vacuum deposition apparatus comprises a valve cell comprising an outer enclosure, an inner tank containing a material to be evaporated, and an inner tank valve of the “drain plug” type. Such a source is, for example, the source of the RIBER Company of the “VCOR” range, marketed under the references “VCOR 110” or “VCOR 300”.

Such a valve cell is made of a material of the PBN (pyrolytic boron nitride) type so that it can be used for the evaporation of corrosive materials such as antimony (Sb), magnesium (Mg), tellurium (Te) or selenium (Se). The inner tank valve is, according to this variant, formed by a control drain plug allowing, in one direction, the flowing of the material to be evaporated, and in the other direction, the filling of the inner tank.

According to a particular embodiment of the invention shown in FIG. 1, the vacuum deposition apparatus **10** also includes pumping means **40** for evacuating the inside of the vacuum deposition chamber **30** to reach a chamber pressure comprised between 10^{-3} and 10^{-12} mbar, typically between 10^{-8} and 10^{-12} mbar.

The pumping means **40** includes first of all a primary pump **41** and a secondary pump **42**, mounted in series.

The primary pump **41** has a primary pump input **41A** and a primary pump output **41B**. The primary pump **41** is here a conventional pump of the diaphragm pump type. Such a primary pump **41** makes it possible to reach vacuum levels with pressures down to 10^{-3} mbar.

As a variant, the primary pump may be for example a scroll pump, a vane pump or a roots pump.

The primary pump output **41B** is connected to an exhaust pipe **14** for discharging all the gases pumped from the vacuum deposition apparatus **10** by the pumping means **40**.

The secondary pump **42** has a secondary pump input **42A** and a secondary pump output **42B** having a substantially identical flow rate. The pressure ratio between the secondary pump output **42B** and the secondary pump input **42A** is here higher than 10000 in conditions of normal operation.

According to a particular embodiment of the invention shown in FIG. 1, the secondary pump **42** is a turbomolecular pump. This turbomolecular pump **42**, which is here a multi-stages turbomolecular pump, allows reaching vacuum levels with pressures in the vacuum deposition chamber **30** comprised between 10^{-3} mbar and 10^{-11} mbar.

The secondary pump **42** is here a “high flow rate” pump, with a pumping speed of the order of 2000 liters per second. The volume of the vacuum deposition apparatus **10** being slightly higher than 2000 liters, the major part of the gases present in the vacuum deposition chamber **30**, for example 99.99% of the gases, is pumped very rapidly, within about 10 seconds.

In the configuration of FIG. 1, the pumping means **40** are mounted on the vacuum deposition apparatus **10** at the level of the vacuum deposition chamber **30**.

As a variant, the pumping means may be mounted on another chamber, for example a loading or unloading chamber, a degassing chamber or a distribution chamber, this other chamber being in fluidic communication with the vacuum deposition chamber of the vacuum deposition apparatus by means of a high-conductance gaseous line.

More precisely, in the embodiment of the invention illustrated in FIG. 1, the pumping means **40** includes a secondary pump valve **43** placed between the vacuum deposition chamber **30** and the secondary pump **42**. This secondary pump valve **43** includes a secondary pump valve input **43A** and a secondary pump valve output **43B**. The secondary pump valve input **43A** is in fluidic communication with the vacuum deposition chamber **30** and the secondary pump valve output **43B** is connected to the secondary pump input **42A**.

Preferentially, the secondary pump valve **43** is here a valve of the “slide gate valve” type.

As a variant, the secondary pump valve may for example be a butterfly valve or a pendulous valve.

When the secondary pump valve **43** is open and when the secondary pump **42** operates, then a vacuum is formed inside the vacuum deposition chamber **30**.

When the secondary pump valve **43** is closed, it isolates the vacuum deposition chamber **30** from the remaining of the pumping means **40**, and in particular from the secondary pump **42**. However, the latter may continue to operate despite the closing of the secondary pump valve **43**.

Indeed, the secondary pump **42** reaches its optimal operating condition after only a certain time, typically after 15 to 30 minutes. Therefore, the secondary pump **42** is not always stopped when the vacuum deposition apparatus **10** operates and when the chamber pressure has reached the set point fixed by the operator.

The pumping means **40** also include a primary pumping valve **44** placed between the secondary pump output **42B** and the primary pump input **41A**. This primary pump valve **44**, when open, allows placing in communication the secondary pump **42** on the one hand, and the primary pump **41** on the other hand.

The pumping means **40** finally include a by-pass valve **45** placed at the output of the secondary pump **42**. More particularly, the by-pass valve **45** is mounted as a “by-pass” of the output of the secondary pump **42B**, the by-pass being made upstream the primary pump valve **44**. This allows in particular, when the by-pass valve **45** is open and when the primary pump valve **44** is closed, conducting the compressed gases coming out of the secondary pump output **42B** toward the output of the by-pass valve **45**. This also allows, when the by-pass valve **45** is closed and when the primary pump valve **44** is open, conducting the compressed gases coming out of the secondary pump output **42B** toward the input of the primary pump **41A**.

As shown in FIG. 1, the vacuum deposition apparatus **10** also comprises a leak detection device **50**.

The following description of the leak detection device **50** and the operation thereof will reveal the advantage of using pumping means **40** as arranged and described hereinabove to detect potential leaks of the inner tank **24**, either at the filling flange **25** or at its inner tank valve **28**.

The leak detection device **50** comprises a helium detector **51** comprising a detector input **51A** and a detector output **51B**.

The leak detection device **50** also comprises gas pumping means (not shown) for sucking up the gases arriving at the detector input **51A** so as to eject them to the detector output **51B**. These gas pumping means are generally weak. They have here a pumping capacity of about 4 liters per second for helium (130 liters per second for nitrogen).

As shown in FIG. 1, the detector input **51A** is connected to the output of the by-pass valve **45** and the detector output **51B** is connected to the exhaust pipe **14**.

The helium detector **51** generally comprises a mass spectrometer (not shown) for measuring the quantity of helium present in the gaseous flow flowing between the detector input **51A** and the detector output **51B**. The helium detector **51** thus delivers a signal representative of the helium flow rate (in liters per second or $\text{l}\cdot\text{s}^{-1}$) passing through it.

The helium detector **51** is here a detector of the ADIXEN Company, marketed under the reference ASM380. The leak detection device **50** equipped with such a helium detector **51** has a helium leak rate detection threshold (or “minimum detectable helium leak rate”) around $5\cdot 10^{-12}$ mbar $\cdot\text{l}\cdot\text{s}^{-1}$.

The leak detection device **50** also includes gas injection means **52, 53** for introducing helium (helium 4) in the vacuum deposition chamber **30**.

As a variant, the gas injection means may for example introduce other light gases such as helium 3 (^3He : isotope of helium 4) or hydrogen (H_2).

In the particular embodiment of the vacuum deposition apparatus **10** according to the invention illustrated in FIG. 1, the gas injection means **52, 53** of the leak detection device **50** also introduce an inert gas in the vacuum deposition chamber **30**, the inert gas forming a gaseous mixture with helium.

As an alternative, the gas injection means may for example be mounted on another chamber in fluidic communication with the vacuum deposition chamber of the vacuum deposition apparatus.

The gaseous mixture introduced here by the gas injection means **52, 53** is a gaseous mixture consisted of helium and dinitrogen (N_2) with a molar fraction of 1000 ppm (ppm=part per million) of helium.

The gaseous mixture of helium and dinitrogen is stored in a bottle **52** containing the gaseous mixture at a pressure comprised between 1 bar and 250 bars and equipped with a low-pressure regulator (not shown).

The gaseous mixture coming out of the bottle **52** is then generally conducted to an injection valve **53** that, if open, allows the flowing of the pressurized gaseous mixture up to the vacuum deposition chamber **30**.

So arranged, the gas injection means **52, 53** allow a gaseous mixture containing at least partially helium to be injected in the vacuum deposition chamber **30**.

The vacuum deposition chamber **30** being here in communication with the inner tank **24** when the inner tank valve **28** is open, the gas injection means **52, 53** thus allow the introduction of the gaseous mixture into the inner tank **24**.

Likewise, as in the particular embodiment of the invention shown in FIG. 1, the chamber volume **31** and the outer enclosure volume **23** communicate with each other, the gas injection means **52, 53** also allow the introduction of the gaseous mixture into the outer enclosure **21**. The gaseous mixture then occupies all the available space, i.e. the outer enclosure volume **23** and the chamber volume **31**, which communicate with each other in the case shown in FIG. 1.

Using such a gaseous mixture, with extremely reduced helium content, the leak detection tests may be performed in higher pressure conditions thus simulating the operation of the vacuum deposition apparatus **10** in normal conditions of use. Therefore, for a same operating pressure, the work is made with a reduced molar fraction of helium, and thus a reduced partial pressure of helium, which limits the problems of saturation of the helium detector **51** during a leak detection test. It is thus possible to perform detection tests as rapidly as with pure helium in “stop” conditions of the vacuum deposition apparatus **10**.

The leak detection device **50** also includes pressure measuring means **54, 54A** inside the vacuum deposition chamber **30**. These measuring means comprise a combined vacuum gauge **54A** placed inside the vacuum deposition chamber **30**.

This combined vacuum gauge **54** generally includes:

- a secondary vacuum gauge of the “Bayard-Alpert ionic gauge” type for measuring chamber pressures comprised between 10^{-10} and 10^{-3} mbar;

- a primary vacuum gauge of the “convection Pirani gauge” type for measuring chamber pressures comprised between 10^{-4} and 1000 mbar.

Such pressure measuring means **54, 54A** thus allow measuring chamber pressures comprised between 10^{-10} and 1000 mbar, and this with a response time of the order of about 10^{-3} second, or even up to about 1 second.

At the end of the injection of the gaseous mixture by the gas injection means **52, 53** in the vacuum deposition chamber **30**,

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when the inner tank valve **28** is open, the secondary pump valve **43** is closed and the gaseous mixture also fills the inner tank **24**, the pressure measuring means **54**, **54A** measure a chamber pressure that is the same as the pressure inside the inner tank **24**, which is in static equilibrium with the vacuum deposition chamber **30**.

The combined vacuum gauge **54A** is generally piloted by a vacuum gauge controller **54** that delivers a signal representative of the pressure (in mbar) inside the vacuum deposition chamber **30**.

As explained hereinabove, when the inner tank **24** is in communication with the vacuum deposition chamber **30** thanks to the opening of the inner tank valve **28**, the gaseous mixture introduced in the vacuum deposition chamber **30** also fills the inner tank **24**, such that the pressure inside the vacuum deposition chamber **30** is equal to the pressure inside the inner tank **24**.

Therefore, the vacuum gauge controller **54** delivers a signal that is also representative of the pressure (in mbar) inside the inner tank **24**.

The leak detection device **50** further includes signal processing means **55** processing the signal relating to the helium flow rate delivered by the helium detector **51** and the signal relating to the pressure in the inner tank **24** delivered by the pressure measuring means **54**, **54A**, to deliver a signal representative of the leak rate of the inner tank **24** and the inner tank valve **28**.

In the particular embodiment shown in FIG. 1, the leak detection device **50** also includes display means **56** connected to the signal processing means **55**, allowing an operator to visualize the result of the leak detection test carried out by the vacuum deposition apparatus **10** according to the invention.

Such display means **56** here comprise a digital screen.

The leak detection device **50** finally preferably includes piloting means **57** controlling, on the one hand, the gas injection means **52**, **53** and, on the other hand, the opening and closing of the inner tank valve **28**, the secondary pump valve **43**, the primary pump valve **44** and the by-pass valve **45**.

The piloting means **57** are linked, on one side, to the signal processing means **55** that provide them with information relating to the leak rates measured in the inner tank **24** and the inner tank valve **28**, and on another side, to four actuators **57A**, **57B**, **57C** and **57D** piloting the injection valve **53**, the secondary pump valve **43**, the by-pass valve **45** and the primary pump valve **44**, respectively.

Furthermore, the piloting means **57** are linked to the inner tank valve actuator **29A** that pilots the inner tank valve **28**.

As described hereinabove for the inner tank valve actuator **29A**, each of the actuators **57A**, **57B**, **57C** and **57D** allows varying and knowing the opening percentage of each of the piloted valves **53**, **43**, **44**, **45**.

The piloting means **57** are in particular programmed to pilot the actuators **57A** and **57D** of the by-pass valve **45** and of the primary pump valve **44** in such a way that:

the primary pump valve **44** is closed when the by-pass valve **45** is open, wherein the compressed gases coming out of the secondary pump output **42B** are then conducted toward the input of the helium detector **51A**, and that:

the by-pass valve **45** is closed when the primary pump valve **44** is open, wherein the compressed gases coming out of the secondary pump output **42B** are then conducted toward the primary pump input **41A**.

Second Embodiment

FIG. 2

FIG. 2 shows a schematic overall view of a vacuum deposition apparatus **200**, according to a second embodiment,

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which comprises a valve cell **220** with an inner tank **224** open in the outer enclosure **21**. The free volume **224A** is thus in direct communication with the outer enclosure volume **23** of the outer enclosure **21**. Therefore, the inner tank **224** comprises no filling flange.

As a variant, it may be provided additional means for heating the inner tank, located outside the outer enclosure.

As another variant, and advantageously, the means for heating the inner tank may be located outside the outer enclosure.

During the heating of the inner tank **224** by the heating means **27**, the material to be evaporated **26** evaporates toward the upper part of the inner tank **224** to fill all the outer enclosure volume **23**, in particular the particular volume **23A** comprised between the first opening **21B** of the outer enclosure **21** and the input port **228A** of the inner tank valve **228** (see FIG. 3). The inner tank valve **228** is thus in fluidic communication with the inner tank **224**.

As shown in FIG. 2, the outer enclosure **21** indeed comprises an enclosure tube **221A** connecting the first opening **21B** to the input port **228A** of the inner tank valve **228**.

Moreover, the inner tank valve **228** forms one of the ends of a valve tube **228C**, the other end being connected to the outer enclosure **21** at the first opening **21B**.

The inner tank valve **228** also comprises an output port **228B** connected to the transfer tube **11** that goes through the second wall **30A** of the vacuum deposition chamber **30** (see FIG. 2).

In this second embodiment, the inner tank valve **228** also makes it possible to isolate the outer enclosure **21** of the vacuum deposition chamber **30**. Indeed, in closed position, the inner tank valve **228** inhibits not only the flowing of the material to be evaporated from the particular volume **23A** toward the chamber volume **31** of the vacuum deposition chamber **30**, but also the placement of the outer enclosure **21** in fluidic communication with the vacuum deposition chamber **30**.

Third Embodiment

FIG. 3

Unlike in the first embodiment of the vacuum deposition apparatus **10** shown in FIG. 1, a weld may exist, for example at the connecting flange, between the outer enclosure and the transfer tube. In this case, the vacuum deposition chamber does not communicate with the outer enclosure and the chamber and outer enclosure volumes are distinct from each other.

Hence, it has been shown in FIG. 3 a schematic overall view of a vacuum deposition apparatus **300**, according to a third embodiment, in which the outer enclosure **21** is also isolated from the vacuum deposition chamber **30**.

Indeed, as shown in FIG. 3, the valve cell **320** is different from the valve cell **20** of the first embodiment (see FIG. 1) in that it comprises means for isolating the outer enclosure **21**.

More particularly, in this third embodiment, it is provided a tight weld **311C** between the outer enclosure **21** and the transfer tube **11**.

Thus, in this particular configuration, the outer enclosure **21** is no longer in fluidic communication with the vacuum deposition chamber **30**, the outer enclosure volume **23** being isolated from the chamber volume **31** thanks to the weld **311C**.

In this third embodiment, the outer enclosure **21** is irrevocably isolated from the vacuum deposition chamber **30** thanks to the weld **311C**.

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This configuration is different from that of the second embodiment of the vacuum deposition apparatus **200** shown in FIG. **2**, in which it is all the same possible to place the vacuum deposition chamber **30** in communication with the outer enclosure **21** through the opening of the inner tank valve **228**.

Leak detection method in the various embodiments of the vacuum deposition apparatus **10**; **200**; **300**

A method according to the invention will now be described, which can be applied by the operator to detect a leak of the inner tank **24**; **224** and/or the inner tank valve **28**; **228**.

This detection method may advantageously be implemented for each of the above-described embodiments of the vacuum deposition apparatus **10**; **200**; **300** (see FIGS. **1** to **3**).

However, it is understood that:

in the first embodiment (see FIG. **1**), the leak detection method according to the invention wholly tests the tightness of the inner tank valve **28** and of the inner tank **24** (i.e. of its filling flange **25**) without being capable of distinguishing the origin of a potential leak;

in the second embodiment (see FIG. **2**), wherein the inner tank **224** cannot have any leak, the leak detection method according to the invention tests only the tightness of the inner tank valve **228**, and

in the third embodiment (see FIG. **3**), wherein the outer enclosure **21** is isolated from the vacuum deposition chamber **30** thanks to the weld **311C**, the leak detection method according to the invention tests also only the tightness of the inner tank valve **28**.

1) Initialization step

The operator first carries out a step of initialization of the vacuum deposition apparatus **10**; **200**; **300**, aiming to measure the leak rate of the residual helium in the vacuum deposition chamber **30**. For that purpose, he performs the following operations:

1a) Preparing the valve cell **20**; **220**; **320** to be tested and the leak detection device **50**

closing the inner tank valve **28**; **228** and placing the filling flange **25** (if present) on the inner tank **24**; **224**;

verifying the tightness of the cell flange **22** and of the vacuum deposition chamber **30**;

closing the secondary pump valve **43**;

opening the primary pump valve **44** and closing of the by-pass valve **45**;

connecting the helium detector **51** to the by-pass valve **45** and powering-up the detector;

preparing the injection means **52**, **53** for injection of the gaseous mixture He/N_2 and draining the injection line.

1b) Evacuating the vacuum deposition apparatus **10**; **200**; **300**

opening the inner tank valve **28**; **228**;

opening the secondary pump valve **43**;

powering-up the primary pump **41** and the secondary pump **42**;

waiting until the pressure in the vacuum deposition chamber **30** is lower than 10^{-5} mbar, preferably lower than 10^{-6} mbar.

1c) Verifying the base signal of the helium detector **51**

closing the primary pump valve **44** and opening the by-pass valve **45**;

measuring the leak rate of the residual helium in the vacuum deposition chamber **30** and waiting for the stabilization of the leak rate of the inner tank **24**; **224** and of the inner tank valve **28**; **228** to a value lower than 10^{-10} mbar·l·s⁻¹;

logging the previous stabilized initial value;

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opening the primary pump valve **44** and closing the by-pass valve **45**.

2) Measurement step

After the initialization step, the operator may proceed to a measurement step aiming to measure the leak rate of the inner tank **24**; **224** and/or the inner tank valve **28**; **228**. For that purpose, he performs the following operations:

2a) Pressurization of the vacuum deposition apparatus **10**; **200**; **300**

verifying that all the temperatures are lower than 800° C.; closing the secondary pump valve **43** and opening the inner tank valve **28**; **228** to place the vacuum deposition chamber **30** in fluidic communication with the inner tank **24**; **224**;

injecting thanks to the injection mean **52**, **53** a helium/nitrogen gaseous mixture at 1000 ppm of molar fraction in helium and filling the vacuum deposition chamber **30** up to chamber pressure set-point comprised between 10^{-4} et 10^{-1} mbar;

logging the static pressure **P0** inside the inner tank **24**; **224** reached after a wait of 1 minute;

2b) Pumping of the vacuum deposition apparatus **10**; **200**; **300**

closing the inner tank valve **28**; **228**;

opening the secondary pump valve **43**;

evacuating the vacuum deposition chamber **30** until the chamber pressure is lower than 10^{-6} mbar;

2c) Measuring the leak rate of the inner tank **24**; **224** and/or the inner tank valve **28**; **228**

closing the primary pump valve **44** and opening the by-pass valve **45**;

measuring the helium leak flow rate by means of the helium detector **51** and calculating, by means of the leak detection device **50**, the leak rate (in mbar·l·s⁻¹) of the inner tank **24**; **224** and/or the inner tank valve **28**; **228**;

logging the final value reached after stabilization.

At the end of these various operations, the operator gains a measure of the leak rate of the inner tank **24**; **224** and/or the inner tank valve **28**; **228** by comparing the stabilized values of the helium leak rate obtained at the initialization step and at the measuring step.

In the case where, before the leak detection method, the operator has measured, by a conventional method during the assembling of the vacuum deposition apparatus **10**; **200**; **300**, the leak flow rate of the inner tank valve **28**; **228**, he can deduce the leak rate of the filling flange **25** based on the measure of the “overall” leak rate of the inner tank **24**; **224** and the inner tank valve **28**; **228**, obtained by the above-mentioned method.

Therefore, the operator may determine the closing torque required for the inner tank valve **28**; **228** to obtain a given tightness level, providing that the filling flange **25** is tight enough. This makes it possible to notify to the user of such a vacuum deposition apparatus **10**; **200**; **300** an efficient closing torque value for the inner tank valve **28**; **228** that is not excessive. Indeed, the inner tank valve, which is generally a metal valve, is more rapidly damaged when closed too tight.

The leak rate measurement is rapid according to the method applied by the operator. Indeed, upon the closing of the primary pump valve **44** and the concomitant opening of the by-pass valve **45**, a tiny part of the gaseous mixture containing only a fraction of pure helium reaches the helium detector **51**, which is therefore not saturated by the helium quantity. The measurement of the leak rate may then be performed very rapidly, typically within 1 second. Moreover, by repeating the procedure hereinafter, the helium detector **51** and thus the leak detection device **50** may be rapidly operative

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again, for example to detect a leak at the level of another inner tank containing another material to be evaporated or to measure the leak rate of the inner tank valve **28**; **228** in other opening or closing positions.

The invention claimed is:

1. A valve-cell vacuum deposition apparatus (**10**; **200**; **300**) including:

at least one valve cell (**20**; **220**; **320**) comprising:

an outer enclosure (**21**),

an inner tank (**24**; **224**), located inside the outer enclosure (**21**) and adapted to contain a material to be evaporated (**26**),

an inner tank valve (**28**; **228**) comprising a valve input port (**28A**; **228A**) in fluidic communication with the inner tank (**24**; **224**) and a valve output port (**28B**; **228B**),

a vacuum deposition chamber (**30**),

a transfer tube (**11**) comprising a tube input port (**11A**) and connected to the valve output port (**28B**; **228B**) of the inner tank valve (**28**; **228**) and a tube output port (**11B**), in such a manner that the tube output port (**11B**) is located inside the vacuum deposition chamber (**30**), the tube output port (**11B**) including an injector (**12**) adapted to generate a jet of vapour of the material to be evaporated (**26**) inside the vacuum deposition chamber (**30**), the vacuum deposition chamber (**30**) being connected to the outer enclosure (**21**) by means of a connecting flange (**13**), and

pumping means (**40**) adapted to evacuate the inside of the vacuum deposition chamber (**30**), the pumping means comprising:

a primary pump (**41**) having a primary pump input (**41A**) and a primary pump output (**41B**), and

a secondary pump (**42**) having a secondary pump input (**42A**) and a secondary pump output (**42B**), the secondary pump output (**42B**) being connected to the primary pump input (**41A**),

characterized in that it includes a leak detection device (**50**) comprising:

gas injection means (**52**, **53**) adapted to introduce helium in the vacuum deposition chamber (**30**),

gas pumping means adapted to pump the helium present in the vacuum deposition chamber (**30**),

a helium detector (**51**) comprising a detector input (**51A**), the helium detector (**51**) delivering a signal representative of the helium flow rate through it,

pressure measuring means (**54**, **54A**) adapted to measure the pressure inside the vacuum deposition chamber (**30**), and

signal processing means (**55**) adapted to process the signal relating to the helium flow rate delivered by the helium detector (**51**) and the signal delivered by the pressure measuring means (**54**, **54A**), to deliver a signal representative of the leak rate of the inner tank valve (**28**; **228**), and in that the pumping means (**40**) also include:

a secondary pump valve (**43**) placed between the vacuum deposition chamber (**30**) and the secondary pump input (**42A**),

a primary pump valve (**44**) placed between the secondary pump output (**42B**) and the primary pump input (**41A**), and

a by-pass valve (**45**) placed between the secondary pump output (**42B**) and the detector input (**51A**), the by-pass being made upstream the primary pump valve (**44**).

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2. The vacuum deposition apparatus (**10**; **300**) according to claim **1**, wherein the inner tank valve (**28**) is located inside the outer enclosure (**21**), with its valve input port (**28A**) connected to the inner tank (**24**).

3. The vacuum deposition apparatus (**10**; **300**) according to claim **1**, wherein the tube input port (**11A**) of the transfer tube (**11**) is located inside the outer enclosure (**21**), with the transfer tube (**11**) going through a first wall (**21A**) of the outer enclosure (**21**) and a second wall (**30A**) of the vacuum deposition chamber (**30**).

4. The vacuum deposition apparatus (**10**) according to claim **1**, wherein the pressure measuring means (**54**, **54A**) deliver a signal representative of the pressure inside the inner tank (**24**).

5. The vacuum deposition apparatus (**10**) according to claim **1**, wherein the outer enclosure (**21**) is in fluidic communication with the vacuum deposition chamber (**30**).

6. The vacuum deposition apparatus (**10**; **200**; **300**) according to claim **1**, wherein the secondary pump (**42**) is a turbomolecular pump.

7. The vacuum deposition apparatus (**10**; **200**; **300**) according to claim **1**, wherein the secondary pump valve (**43**) is a slide gate valve.

8. The vacuum deposition apparatus according to claim **1**, wherein the secondary pump valve is a butterfly valve or a pendulous valve.

9. The vacuum deposition apparatus (**10**; **200**; **300**) according to claim **1**, wherein the gas injection means (**52**, **53**) of the leak detection device (**50**) are also adapted to introduce an inert gas in the vacuum deposition chamber (**30**), the inert gas forming a gaseous mixture with helium.

10. The vacuum deposition apparatus (**10**; **200**; **300**) according to claim **1**, wherein the leak detection device (**50**) also comprises piloting means (**57**) adapted to control the gas injection means (**52**, **53**) and the opening and closing of the inner tank valve (**28**; **228**), the secondary pump valve (**43**), the primary pump valve (**44**) and the by-pass valve (**45**).

11. The vacuum deposition apparatus (**10**) according to claim **1**, wherein the signal processing means (**55**) deliver a signal representative of the leak rate of the inner tank valve (**28**) and the inner tank (**24**).

12. The vacuum deposition apparatus (**10**; **300**) according to claim **11**, wherein the inner tank valve (**28**) is located inside the outer enclosure (**21**), with its valve input port (**28A**) connected to the inner tank (**24**).

13. The vacuum deposition apparatus (**10**; **300**) according to claim **11**, wherein the tube input port (**11A**) of the transfer tube (**11**) is located inside the outer enclosure (**21**), with the transfer tube (**11**) going through a first wall (**21A**) of the outer enclosure (**21**) and a second wall (**30A**) of the vacuum deposition chamber (**30**).

14. The vacuum deposition apparatus (**200**; **300**) according to claim **1**, wherein there are provided isolation means (**228**; **311C**) adapted to isolate the outer enclosure (**21**) from the vacuum deposition chamber (**30**).

15. The vacuum deposition apparatus (**200**) according to claim **14**, wherein the isolation means comprise the inner tank valve (**228**).

16. The vacuum deposition apparatus (**300**) according to claim **14**, wherein the isolation means comprise a weld (**311C**) between the outer enclosure (**21**) and the transfer tube (**11**).

17. A method for detecting a leak in a valve-cell vacuum deposition apparatus (**10**) by deducing a leak rate, the vacuum deposition apparatus (**10**) including:

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at least one valve cell (20) comprising:
 an outer enclosure (21),
 an inner tank (24; 224), located inside the outer enclosure (21), wherein the inner tank (24; 224) contains a material to be evaporated (26),
 an inner tank valve (28; 228) comprising a valve input port (28A; 228A) in fluidic communication with the inner tank (24; 224) and a valve output port (28B; 228B),
 a vacuum deposition chamber (30), and
 a leak detection device (50),
 the leak detection method being characterized in that it includes:
 an initialization step wherein:
 the inside of the vacuum deposition chamber (30) is evacuated, and
 an initial leak flow rate in the vacuum deposition chamber (30) is measured by means of the leak detection device (50), and
 a measurement step wherein:
 the inner tank valve (28; 228) is opened to place the vacuum deposition chamber (30) in fluidic communication with the inner tank (24; 224), and
 a gaseous mixture is injected in the vacuum deposition chamber (30), and
 a pressure P0 inside the inner tank (24; 224) is measured, and

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the inner tank valve (28; 228) is closed, and
 the inside of the vacuum deposition chamber (30) is evacuated down to a predetermined pressure in the vacuum deposition chamber (30), and
 the vacuum deposition chamber (30) is placed in communication with the leak detection device (50), and
 a final leak flow rate in the vacuum deposition chamber (30) is measured by means of the leak detection device (50), and

a leak rate of the inner tank valve (28; 228) is deduced from the final leak flow rate and the initial leak flow rate.

18. The leak detection method according to claim 17, wherein the inner tank valve (28) of the vacuum deposition apparatus (10; 300) is located inside the outer enclosure (21), with its valve input port (28A) connected to the inner tank (24).

19. The leak detection method according to claim 17, wherein, at the end of the measurement step, the leak rate of the inner tank valve (28; 228) and the inner tank (24; 224) is deduced.

20. The leak detection method according to claim 19, wherein the inner tank valve (28) of the vacuum deposition apparatus (10; 300) is located inside the outer enclosure (21), with its valve input port (28A) connected to the inner tank (24).

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